

Cooling and coke deposition of hydrocarbon fuel with catalytic steam reforming

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ABSTRACT

The strong endothermic reaction of hydrocarbon-fuel catalytic steam highlights its potential for use in high-temperature wall cooling. The cooling capacity and cracking-controlled coke of hydrocarbon fuel have been compared experimentally with and without a catalytic steam reforming reaction under supercritical pressure conditions, and the interaction effects between the thermal cracking and catalytic reforming reactions were analyzed. The results show that the hydrocarbon fuel catalytic steam reforming reaction can not only significantly improve the total heat sink and fuel conversion, but also reduce the coke deposition arising from the thermal cracking process. The chemical heat sink of the catalytic steam reforming reaction is higher than that of thermal cracking, especially in the high-temperature range. At temperatures below 500 °C, catalytic steam reforming provides a boost to the fuel compounds and turns them into small-molecular gas-phase products such as hydrogen and carbon monoxide. The catalytic reforming and thermal cracking reactions occur simultaneously at temperatures above 500 °C. The catalytic reforming reaction can lower the degree of thermal cracking and inhibit the polymerization of small molecules produced by cracking to aromatics, consequently resulting in less coke deposition.

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1. Introduction

The heat load on the surface of an engine increases sharply with an increased flight Mach number, and hydrocarbon fuel regenerative cooling has been considered as an effective form of thermal protection to solve the aerodynamic heating problems associated with hypersonic flight conditions. The lack of sufficient endothermic heat sink capability from aviation kerosene, however, has limited the development of regenerative cooling systems. Thermal protection systems would require 4 MJ/kg of total heat sink at Mach number 8, which represents a heat sink capability that surpasses the maximum value of most jet fuels [1].

A number of studies have been conducted with the aim of improving the heat sink capability of hydrocarbon fuels, including (1) enhancing the upper limit of the available operating temperature range using a hydrocarbon hydrogenation process or an additive package [2–4]; (2) developing endothermic fuels by making changes to the fuel compositions that control the properties of the hydrocarbon fuel [5–7]; and (3) generating endothermic reactions in the hydrocarbon fuel under catalytic dehydrogenation and cracking conditions [8,9].

Although the above measures, especially catalytic dehydrogenation and cracking methods, can generate a higher heat sink from fuel, the high cost of the platinum catalyst as well as the formation of a large amount of coke deposition has limited the use of these techniques.

Further development is needed to enhance the fuel heat sink capacity of jet fuels while at the same time to mitigate the carbon deposition from thermal cracking.

As one of endothermic catalytic reactions that can increase the cooling capacity, achieve high levels of hydrogen production and minimal coke deposition, the catalytic steam reforming of hydrocarbon fuel has recently drawn significant attention [10–12]. Catalytic steam reforming technology, originating in the chemical industry [13], is used to produce hydrogen and ammonia, and subsequently has been developed in the fields of fuel cell [14,15], internal combustion engines [16], and gas generators [17]. In recent years, this technique has been investigated for thermal protection of hypersonic flight vehicle. Korabelnikov et al. [18–20] studied the catalytic conversion and output characteristics of steam reforming for methane, heptane, and decane. It is found that the reaction system with catalytic reforming could lead to a fuel having a longer working life even in a thermal cracking regime. Hou et al. [21] investigated the heat sink capacity and conversion of a catalytic steam reforming reaction for aviation kerosene. However, the quantitative measurement of coke deposition under the catalytic reforming condition and the interaction between thermal cracking and catalytic steam reforming reactions have not been studied previously.

The objective of this work is to provide new insights into the cooling capacity and coke deposition behaviors under the coupling interaction between thermal cracking and steam reforming reactions. To do so, we have conducted a series of experimental studies of the catalytic steam reforming based on a specific hydrocarbon fuel. The thermal

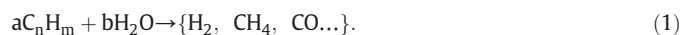
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cracking and coke deposition behaviors as well as the heat sink performances of the fuel with and without the catalytic steam reforming reaction have been compared and analyzed. Results on the coupling interactions between the thermal cracking and steam reforming reactions were obtained.

2. Principle and experimental systems

The heat sink capacity of a hydrocarbon fuel can be improved by the catalytic steam reforming reaction. In essence, the C–H bonds in the fuel and the H–O bonds in steam are broken and reformed into hydrogen, carbon monoxide, and low molecular weight hydrocarbons on the surface of the catalyst:



This reaction serves as a thermal protection component since it is an endothermic process. It also generates flammable gaseous species, such as hydrogen, carbon monoxide, and low-molecular-weight hydrocarbon compounds, which effectively shorten the ignition delay of the fuel in the kerosene-fueled supersonic combustor.

The test apparatus that was used under supercritical pressure conditions is shown in Fig. 1. High purity nitrogen gas was used to remove the residuals before and after the test process. Chinese kerosene was used as hydrocarbon fuel. The critical pressure and temperature are 2.459 MPa and 404 °C, respectively. Cycloalkanes are major components of kerosene and constitute up to 49% of the fuel in mass. Water and kerosene, together with 0.3% emulsifier, were fed into the fuel tank in advance of the test process and then mixed to form an emulsion fuel using a static in-pipe mixer. The fuel itself was pressurized into a pre-heater and the reactor using a metering pump following a filtration treatment process. A Coriolis mass flowmeter was used to measure and control the flow rate of the liquid fuel. The capacity of the flowmeter is 10 MPa, and the flow rate limit is 3 g/s. Its measurement resolution is $\pm 0.2\%$. As a result, the fuel supply can be delivered in a stable and uniform way to the subsequent heating and sampling sections.

The fuel was then heated using electric power in a pre-heater and a plate reactor. The fuel at the outlet can be heated to the maximum temperature of 700 °C, surpassing the critical temperature 404 °C. In the preheater, a pipe coil with an inner diameter of 2 mm and a 1 mm wall was coiled around the electric-heating rod and inserted into the small-scale heating furnace. The plate reactor was used to verify the cooling capacity of the high-temperature plane surface via the endothermic reaction, as shown in Fig. 2. The plate reactor was made of high-temperature alloy stainless steel and consisted of five loops with a total length of 2883 mm and a rectangular cross-sectional dimension of 4×2 mm. In addition to the fuel temperature measurements at the

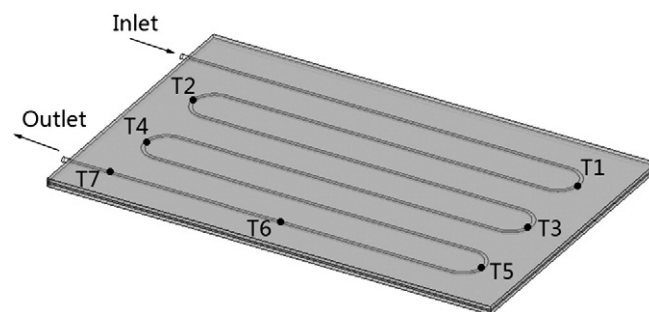


Fig. 2. Schematic diagram of the reactor.

reactor inlet and exit, seven thermocouples were used to measure the outer-wall temperatures along the flow path of the reactor. An inexpensive nickel-based reforming catalyst from Sinopec Qilu Company was coated onto the inner wall of the reactor by means of plasma spraying using a ceramiclike binder, and the thickness of the catalytic layer was controlled within 0.2 mm. Nickel element as an active component is added in the carrier of calcium aluminate using an impregnation method. Potash is used as an additive in the reforming catalyst for the resistance to carbon formation. The two sides of the reactor were covered with electric-heating boards. The heating flux was simulated by gradually regulating the output voltage of the power.

The plate reactor was heated electrically and most of the heat was absorbed by fuel. A small portion of the heat, however, was dissipated to the environment by means of heat loss. The energy equation of the experimental system could be expressed as follows:

$$P_{in} = q_m \Delta h_T + P_{loss} \quad (2)$$

where P_{in} is the input power of the electrical heater, q_m is the mass flow rate of the fuel, Δh_T is the total heat sink per unit fuel of mass, and P_{loss} is the system heat loss to the environment that could be minimized through insulation and be obtained through calibration without a fuel flow. Using the current setup, the heat loss was measured to be in the range of 13.4–16.5% of the total power input, depending on the reactor's wall temperature and the environment.

The total heat sink represents the endothermic ability of the fuel and takes both the physical heat sink (Δh_p) and the chemical heat sink (Δh_c) into account. The physical heat sink can be calculated as a function of the fuel temperature measured at the reactor inlet (T_{in}) and exit (T_{out}). The fuel composition is assumed unchanged for the calculation of the sensible enthalpy. The constant-pressure specific heat can be computed according to the procedure described by Hu [22]. The chemical heat sink Δh_c is the endothermic heat sink of the fuel thermal cracking

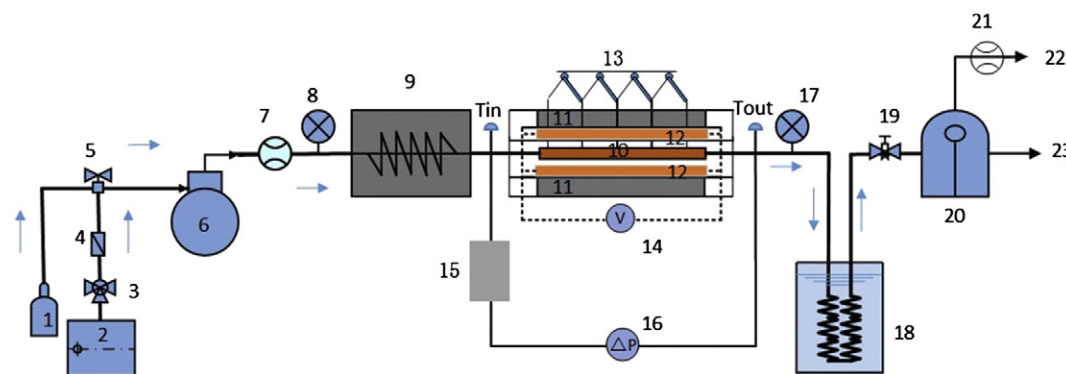


Fig. 1. Experimental setup. 1. High purity nitrogen; 2. fuel tank; 3. online static mixer; 4. filter; 5. switch valves; 6. metering pump; 7. mass flow meter; 8. pressure gauge; 9. pre-heater; 10. reactor; 11. insulating layer; 12. heating plates; 13. temperature measurement; 14. voltage regulators; 15. overheat protection; 16. differential pressure transmitter; 17. pressure gauge; 18. condenser; 19. backpressure regulator; 20. gas-liquid separator; 21. gas flow meter; 22. Gaseous products; 23. liquid products.

and/or catalytic reactions, which is difficult for direct measurement due to the complicated reaction process. Its value is obtained as follows:

$$\Delta h_c = \Delta h_T - \Delta h_p \quad (3)$$

The supercritical operating pressures of 3 MPa and 5 MPa were controlled using the back pressure valve, respectively. The reaction products were transported to the gas–liquid separator after cooling. The gaseous and liquid residual products of the reactions were sampled and analyzed by gas chromatography (GC SP-3430, Beijing Analysis Instrument Co., Ltd) and a gas chromatography–mass spectrometer (GC–MS DSQ, Thermo Fisher Scientific Inc.), respectively.

The measurement of coke deposition was carried out using a differential pressure flow resistance method. A Rosemount 3051s differential pressure transmitter with accuracy up to 0.025% was used to measure and track-in-time the reactor pressure drop, which is indicative of coke deposition. Based on a coke density of 1 g/cm³ [23], the coke deposition is determined by the variation of equivalent hydraulic diameter derived from the pressure drop before and after the hot experiments.

3. Results and discussion

Two cases were experimentally investigated and compared. Case 1 referred to as 10% water + 90% kerosene with catalyst represents an emulsified fuel composed of a 90% mass fraction of kerosene fuel and a 10% mass fraction of water that was premixed with 0.3% of an emulsifier. The reaction process in the catalytic reactor section involved a fuel thermal cracking reaction as well as a catalytic steam reforming reaction. Case 2 referred to as 100% kerosene without catalyst only has a thermal cracking reaction and no catalytic steam reforming reaction. For each case, two tests were conducted under the supercritical operating pressures of 3 and 5 MPa, respectively. The flow rates of all of the tests were kept in 1 g/s.

3.1. Heat sink

As shown in Fig. 3, the total heat sink values of the 10% water + 90% kerosene with the catalytic steam reforming reaction are higher than those of the 100% kerosene without the catalyst. The difference of the total heat sink between the catalytic steam reforming and thermal cracking reactions increases with an increase in fuel temperature. The total heat sink of 10% water + 90% kerosene is 0.36 MJ/kg higher than that of the 100% kerosene under a pressure of 3 MPa at 420 °C. The difference increases to 1.5 MJ/kg when the temperature reaches 650 °C. The total heat sink under a pressure of 3 MPa is slightly higher than that observed at a supercritical pressure of 5 MPa. Similar variation trend can be seen from the previous work [21,24]. It is illustrated that

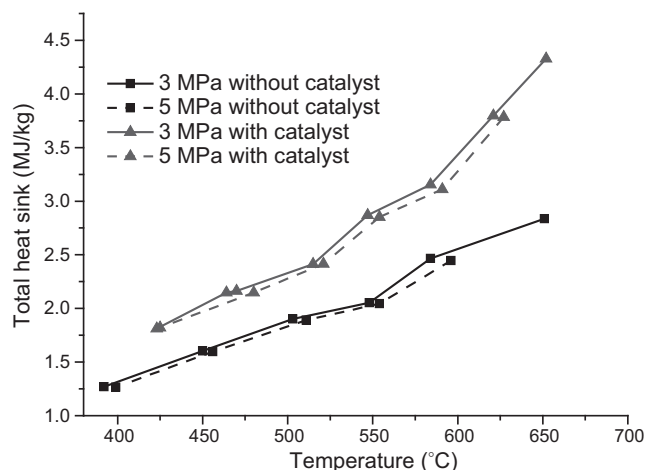


Fig. 3. Total heat sink under supercritical conditions.

the increase in pressure inhibits the catalytic reforming reaction and promotes the thermal cracking reaction. The heat sink decreases during the range of relatively low temperature as a result of the inhibition of catalytic reforming reaction. For the range of relatively high temperature, the heat transfer becomes weak because of the increased coke deposition caused by the enhancement of thermal cracking reaction with an increase in pressure.

Steam can not only improve the physical heat sink properties of a fuel but also promote the chemical heat sink of a fuel through an endothermic reaction in the presence of a catalyst. To determine the contribution made by water to the whole chemical heat sink of fuel during the catalytic steam reforming reaction, we compared the total heat sink between the 10% water + 90% kerosene with the catalytic steam reforming reaction and the weighting of the 90% kerosene without the catalyst plus 10% water enthalpy (shown in Fig. 4(a)). The water enthalpy changes slightly at different temperatures, and it is 0.33 MJ/kg at 426 °C, which becomes 0.39 MJ/kg at 680 °C and accounts for only 15% of the total heat sink. The total heat sink of the 10% water + 90% kerosene with the catalytic reforming reaction is higher than the weighed total heat sink at all temperatures. The difference between the two heat sinks represents the chemical heat sink generated by the catalytic reaction, which is found to increase with increasing temperature. The chemical heat sink of the catalytic steam reforming reaction makes up a quarter of the total heat sink at a temperature of 680 °C. The chemical heat sink of the catalytic steam reforming reaction increases by 470% from 430 to 684 °C, whereas the water enthalpy increases only by 17%. The endothermic process of the catalytic steam reforming reaction therefore has a strong positive effect on promoting the fuel heat sink.

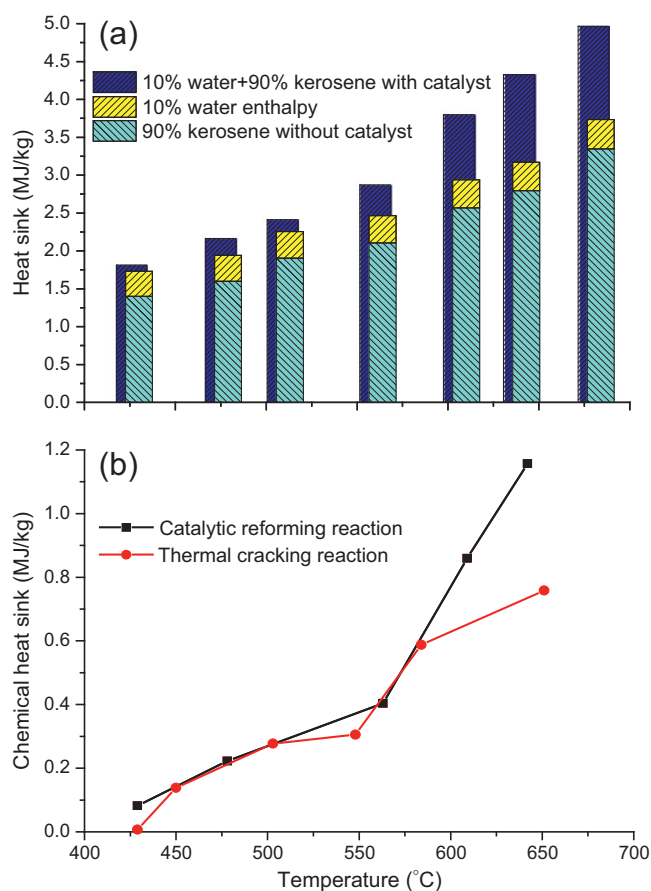


Fig. 4. Heat sink comparisons under different conditions. (a) Total heat sink; (b) chemical heat sink.

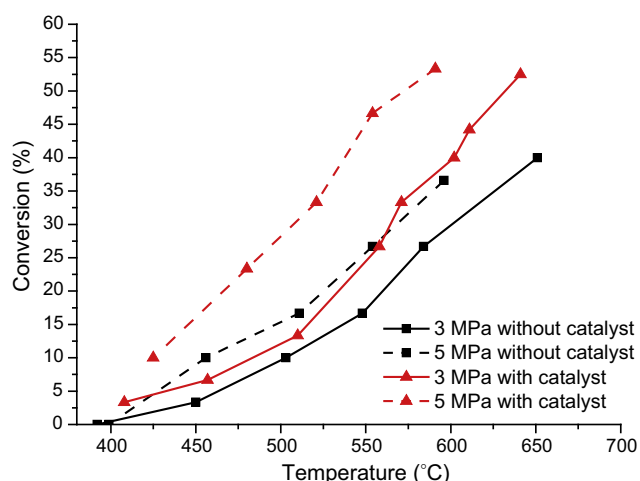


Fig. 5. Fuel conversion under different conditions.

To distinguish the chemical heat sinks generated from the catalytic steam reforming and the thermal cracking reactions, the chemical heat sinks of the catalytic steam reforming and the thermal cracking reactions are compared in Fig. 4(b). The chemical heat sink of the catalytic reforming reaction is calculated from the difference between the total heat sink of the 10% water + 90% kerosene with the catalyst and the weighted sum of the 10% water enthalpy and 90% kerosene without the catalyst. The chemical heat sink of the thermal cracking reaction is obtained from the 90% kerosene without the catalyst. The chemical heat sink of the fuel increases with increasing temperature. The thermal cracking reaction of kerosene increases sharply from a temperature of 550 °C, which results in an increase in the amount of thermal cracking gas-phase products. At the same time, the catalytic steam reforming reaction also plays an important role in enhancing the chemical heat sink and there are interactions between the catalytic reforming and thermal cracking reactions.

The chemical heat sink of the catalytic reforming reaction is similar to that of the thermal cracking reaction below 550 °C, but is higher at fuel temperatures in the range of 550–650 °C. The results indicate that with a water content of only 10%, the catalytic steam reforming reaction of kerosene can supply an additional heat sink similar to that of the thermal cracking reaction at low temperatures and far greater than the heat sink provided by the thermal cracking reaction at higher temperatures. These results show that the improvement in the total heat sink caused by the 10% water content kerosene with the catalytic steam reforming reaction occurs predominantly as a consequence of the endothermic quantity of the steam reforming reaction.

3.2. Reaction conversions and coke deposition

The fuel conversion is defined as the reacted fuel mass flowrate divided by the total fuel mass flowrate. The reacted fuel mass flowrate can be obtained by subtracting the mass flowrate of residual liquid products from the total fuel mass flowrate. As Fig. 5 shows, the conversion of the 10% water + 90% kerosene with the catalytic reforming reaction is higher than that of the 100% kerosene without the catalyst at all of the temperatures and pressures tested. The conversion increases significantly as the temperature of the reaction increases. No conversion is observed for the 100% kerosene without the catalyst at 400 °C. When the temperature is increased to 650 °C, the conversion reaches 40% under the 3 MPa conditions, whereas a temperature of 591 °C gives a conversion rate of 36.7% under the 5 MPa conditions. The conversion of the catalytic steam reforming reaction is only slightly higher than that of the thermal cracking from 400 °C. As the temperature increases, the catalytic steam reforming reaction improves significantly in terms of its fuel conversion, which is 15% higher than that of the fuel under the

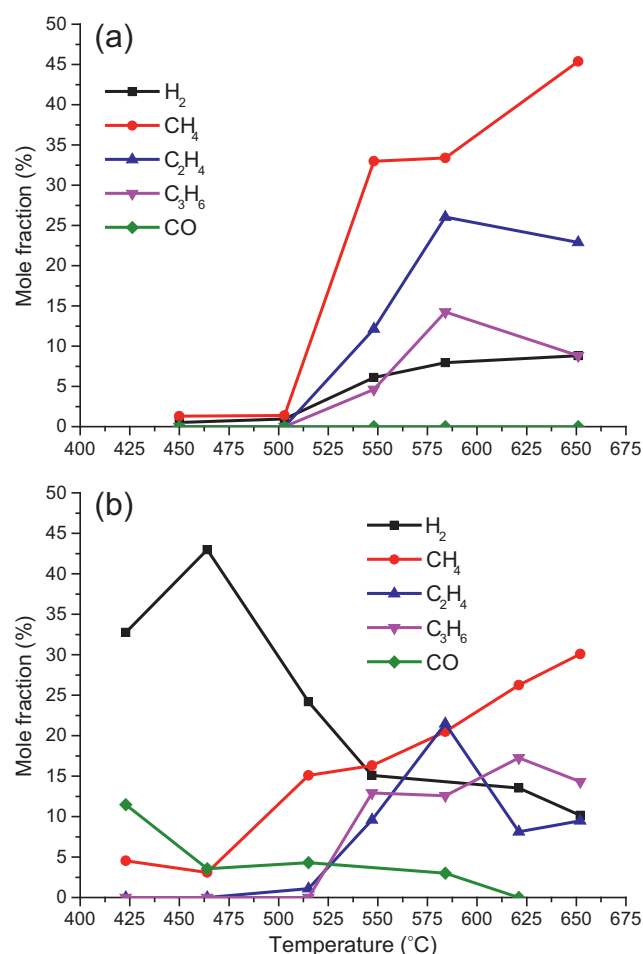


Fig. 6. Comparison of gaseous products with and without catalytic reforming. (a) Without catalytic reforming; (b) with catalytic reforming.

thermal cracking conditions at 650 °C; however, the difference in the conversion observed in this case is only within 5% for temperatures in the range of 400–500 °C. The conversion of the fuel under the 5 MPa conditions is higher than that under the 3 MPa conditions. The increase in the operating pressure effectively accelerates the conversion process.

As shown in Fig. 6 and listed in Table 1, the mole fractions and yields of hydrogen with a catalytic reforming reaction are higher than those without a catalytic reforming reaction, especially at the relatively low temperatures. There is no carbon monoxide and less than 10% hydrogen under thermal cracking conditions without catalytic reforming.

Table 1
Gaseous product yields with and without catalytic reforming.

Temperature (°C)	Gas yield (L/kg)				
	H ₂	CH ₄	C ₂ H ₄	C ₃ H ₆	CO
<i>Without catalytic reforming</i>					
450	0.006	0.017			
503	0.05	0.072			
548	0.54	2.9	1.07	0.41	
584	0.85	3.58	2.79	1.53	
651	1.39	7.14	3.6	1.39	
<i>With catalytic reforming</i>					
423	0.57	0.079			0.2
464	1.94	0.14			0.16
515	2.08	1.29	0.09	0	0.37
547	1.81	1.96	1.15	1.55	0.36
621	2.25	4.37	1.35	2.87	
652	1.97	5.83	1.84	2.77	

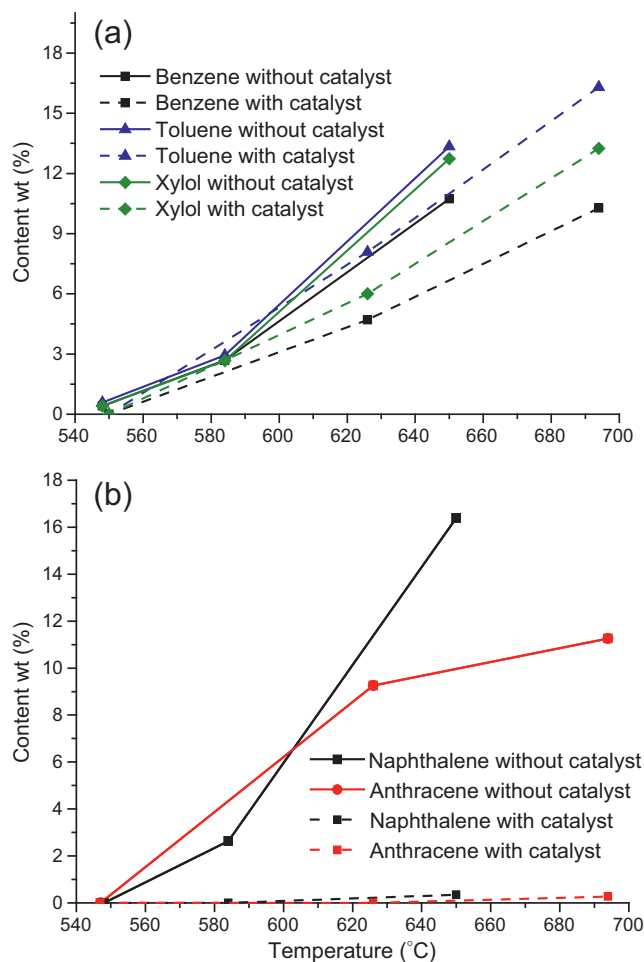


Fig. 7. Comparison of aromatic and PAH products with and without catalytic reforming. (a) Aromatic products; (b) PAH products.

Catalytic reforming generates 33% hydrogen, 12% carbon monoxide, and 5% methane at 425 °C. The concentration of hydrogen with the catalyst reaches a maximum of 42.98% at a temperature of 464 °C, and then gradually decreases with increasing temperature because of an increase in other gaseous products, such as methane, ethene, and propylene, resulting from the thermal cracking reaction. Above 500 °C methane, ethene and propene gradually become the major products. The content of methane without catalytic reforming is obviously higher than that with catalytic reforming. Methane resulting from the thermal cracking process can react with steam and produce hydrogen under catalytic conditions, which causes the rate of methane production to fall under the catalytic conditions. The proportion of ethene in the gaseous products from thermal cracking without catalytic reforming is higher than that from thermal cracking with a catalytic steam reforming reaction. Especially above 575 °C, the proportion of ethane with catalytic reforming decreases significantly.

The variations in the gaseous products indicate the change in the reaction process over the range of temperatures studied. Hydrogen, carbon monoxide, and methane are formed as the major products at

temperatures below 500 °C. These results indicate that the catalytic reforming reaction effectively controls the whole reaction process. The thermal cracking of fuel then starts to take place simultaneously as the temperature further increases, and the proportion of small molecule hydrocarbon products starts to increase. An obvious difference caused by the catalytic steam reforming reaction is that the proportion of methane is much lower than that observed in the thermal cracking reaction. The catalytic steam reforming reaction not only has an adverse impact on the formation of methane but also consumes a certain amount of methane through the steam reforming reaction. It is concluded that the catalytic steam reforming reaction inhibits the thermal cracking of the fuel.

During the process of fuel thermal cracking, the content of ethene significantly increases with an increase in temperature. As a result, butadiene can be formed via an addition reaction and, in a subsequent step, benzene can be formed by the Diels–Alder coupling of butadiene and ethene. Because ethene is the major gaseous product of thermal cracking at high temperatures, it is relatively easy to form aromatic compounds such as benzene and polycyclic aromatic hydrocarbons (PAH), leading to the formation of coke. As shown in Fig. 6, the lower amounts of ethene produced when the catalytic steam reforming reaction takes place decrease the formation of aromatics.

As illustrated in Fig. 7, the total amounts and rates of formation of aromatics arising from thermal cracking without catalytic reforming are higher than those found when catalytic reforming also takes place. Toluene is the most abundant followed by xylene and then benzene. The contents of PAH from thermal cracking without catalytic reforming reaction are far higher than those observed when catalytic steam reforming also takes place. Almost no PAH formation is observed for thermal cracking plus catalytic reforming.

The variation of equivalent diameters and their variation percentage of cooling channel are obtained by measuring the pressure difference to calculate the coke deposition. As Table 2 lists, the amount of coke deposition increases with an increase in temperature. The extent of coke deposition with catalytic reforming is obviously lower than that without catalytic reforming. The variation percentage in the equivalent diameter changes from 5.5% to 0.17% around 600 °C and from 6.6% to 4.5% around 684 °C when a reforming catalyst is used. Coke deposition decreases by 97% around 600 °C and 32% around 684 °C. The catalytic reforming reaction carried out with a water content of 10% can significantly reduce coke formation.

4. Conclusions

The catalytic steam reforming reaction for endothermic fuel can significantly promote the total and chemical heat sinks, which are higher than those of the thermal cracking reaction. The chemical heat sink of the catalytic steam reforming reaction increased by 470% from 430 °C to 684 °C, which represents a quarter of the total heat sink at 684 °C. This increase in the heat sink of the catalytic steam reforming reaction becomes even larger as the temperature further increases.

The fuel conversion of the catalytic reforming reaction is much higher than those of the reaction conducted without the catalytic reaction, which suggests that the catalytic steam reforming reaction provides a stronger reaction process. Increases in the supercritical pressure lead to improvements in the conversion of the fuel.

Table 2
Coke deposition and equivalent diameter variation.

Sample	Temperature (°C)	Equivalent diameter variation (μm)	Equivalent diameter variation percentage (%)	Coke deposit (mg/cm ²)
100% kerosene without catalyst	600	177.45	5.5	6.36
100% kerosene without catalyst	683	211.29	6.6	7.19
10% water + 90% kerosene with catalyst	604	5.458	0.17	0.1931
10% water + 90% kerosene with catalyst	684	144.379	4.5	4.9357

The catalytic endothermic reaction of steam reforming can take place from 400 °C and produce a significant quantity of hydrogen and carbon monoxide from large-molecular hydrocarbons at low temperatures. Thermal cracking and catalytic reforming reactions both occur at temperatures in excess of 500 °C. More hydrogen is produced by catalytic steam reforming than the case with thermal cracking alone. A lower ethene production generated by catalytic reforming restricts the formation of aromatics such as benzene, toluene, and xylene. The catalytic steam reforming reaction can reduce the rate of formation of coke precursors, i.e., PAHs, resulting in the obvious decrease of coke deposition.

Acknowledgments

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